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John C. Martin, Jr.
Senior Patent Counsel

November 21, 1995

John Kollar
President
Redox Technologies Inc.
6 Spencer Court
Wyckoff, NJ 07481

Re: Redox's EG Technology

Dear Mr. Kollar:

I acknowledge receipt of your October 31 letter relating to Redox's EG Technology.

Following our October 31 meeting, we reviewed earlier technical suggestions and enclose for your information a copy of Technical Suggestion 81-73 signed October 16, 1981 titled "Improved Process for the Production of Ditertiary Butyl Peroxide." Preparation of DTBP is described by the reaction of either TBA or isobutylene using tertiary butyl hydroperoxide (TBHP) and an ion exchange acid resin catalyst. The first paragraph, second typed page of the Technical Suggestion states that "a large pore size resin would be preferable." As you know, large pore size resins are known as macroreticular resins.

Therefore, this Technical Suggestion meets the paragraph 6(b) exception to confidentiality of the April 23, 1987 agreement in that the information disclosed to ARCO Chemical Company was in its possession prior to receipt from Redox.

As I stated at our October 31 meeting, we are willing, at your request, to provide affidavits from all named inventors of our 5,371,298 patent stating that they did not have access to any of Redox's technology (i.e., their invention was independently developed from your information). See paragraph 6(d) of the April 23, 1987 agreement.

Some of us believe that using macroreticular resins for this reaction is in the publicly available literature such as early Rohm & Haas Company product bulletins describing uses of macroreticular resins. To date, such literature has not been uncovered.

In any event, the information already provided to you by this letter and at our October 31 meeting clearly eliminates any possible claim by Redox against ARCO Chemical Company under the April 23, 1987 agreement.

Sincerely.

John C. Martin, Jr.

cc: W. J. Klingebiel

Enclosure

ARCO Chemical Company

Technical Suggestio

T. S. No. 81-13 Date Assigned 10/16/81

(1) Title of Invention Improved Process for the Production of Ditertiary Butyl Peroxide

(2) - (3) - (4) - (5)

Ditertiary butyl peroxide (DTBP) is a specialty product used primarily as an organic peroxide catalyst or catalyst intermediate. It is commercially produced by the reaction of pure t-butanol (TBA) or isobutylene with hydrogen peroxide in the presence of concentrated sulfuric acid:

$$2(CH_3)_3COH + H_2O_2 \xrightarrow{70\%\ H_2SO_4} (CH_3)_3COOC(CH_3)_3 + H_2O$$

The process requires refrigeration to control the reaction isotherm (ΔH = 98,000 Btu/lb mole) at or below 15°C thus reducing side reactions and TBA dehydration. The product mixture is quenched with excess water to promote phase separation. The organic phase, consisting of mostly DTBP, is washed with dilute alkali solution and the aqueous acid phase is usually neutralized and discarded.

When excess hydrogen peroxide is used in the above reaction, a mixture tert-butyl hydroperoxide and DTBP is obtained.

An alternative process, that has been experimentally demonstrated and recently evaluated (1), substitutes tert-butyl hydroperoxide for the hydrogen peroxide:

Project No. _____

(6) Test data and original references may be found on the following notebook pages 187434, 187435, _____

Inventor(s) Signature	Date signed	Date of Conception
E. A. Hazbun E. A. Hazbun	10/13/81	
	Date signed	Date of Conception
Signature of Witness	Date signed	Date first disclosed to me
V. M. Chong	10/15/81	
Signature of Witness	Date signed	Date first disclosed to me
J. A. Cahill	10/15/81	

IMPORTANT

USE PEN OR TYPEWRITER - Suggested form for disclosure: (1) Title of invention, (2) State the practices prior to your invention, the problems and disadvantages in connection with these prior practices, and how your invention solves these problems, (3) Your solution or invention - describe fully in prose, with drawings, diagrams, and structural formulae, when appropriate, (4) Give test results illustrating merits of your invention, (5) Describe principal advantages over prior practice which are realized by your invention, (6) List reference pages of laboratory notebook and the persons to whom the invention has been disclosed, (7) Names of inventor(s) and witnesses are recorded on each sheet of the disclosure, (8) Cross out unused space, (9) Forward disclosure to Patent Department.

ARCC-45

(1) Title of Invention: Improved Process for the Production of Ditertiary Butyl Peroxide

(2) - (3) - (4) - (5)

$$(CH_3)_3COH + (CH_3)_3COOH \xrightarrow[H_2SO_4]{70\%} (CH_3)_3COOC(CH_3)_3 + H_2O$$

This also requires refrigeration and acid neutralization. The estimated transfer price via this process at the 3 MM lbs/yr scale is $1.16/lb for pure DTBP.

Recent preliminary testing of DTBP as a cetane improver for diesel fuels (2) have shown the following encouraging results:

V %	Cetane #
0	44.8
3	56.8
5	67.8

Such a performance may create a large volume application for DTBP. if it can be manufactured at low cost.

To allow continuous production at low cost, I propose that DTBP be produced by the reaction of TBHP and TBA over an ion exchange acid resin catalyst at a temperature sufficiently elevated to permit cooling with cooling tower water obviating the need for refrigeration.

Project No. _____

(6) Test data and original references may be found on the following notebook pages 187435, 187436

Inventor(s) Signature	Date signed	Date of Conception
E. A. Hazbun E. A. Hazbun	10/13/81	
	Date signed	Date of Conception
Signature of Witness: V. M. Chong	Date signed: 10/15/81	Date first disclosed to me
Signature of Witness: J. A. Cahill	Date signed: 10/15/81	Date first disclosed to me

IMPORTANT

USE PEN OR TYPEWRITER - Suggested form for disclosure: (1) Title of invention, (2) State the practices prior to your invention, the problems and disadvantages in connection with these prior practices, and how your invention solves these problems, (3) Your solution or invention - describe fully in prose, with drawings, diagrams, and structural formulae, where appropriate, (4) Give test results illustrating merits of your invention, (5) Describe principal advantages over prior practice which are contained by your invention, (6) List reference pages of laboratory notebook and the persons to whom the invention has been disclosed, (7) Names of inventor(s) and witnesses are recorded on each sheet of the disclosure, (8) Cross out unused space, (9) Forward disclosure to Patent Department.

(1) Title of Invention: Improved Process for the Production of Ditertiary Butyl Peroxide

(2) - (3) - (4) - (5)

Based on past experience with acid resin catalysts for MTBE and SBA production, these catalysts tend to yield more moderate reaction rates at higher selectivity than concentrated sulfuric or other mineral acid. A large pore size resin would be preferable in this instance.

The reactor may be a multistage packed bed with interstage cooling or a multitube heat exchange design.

Since the purity requirements for a diesel fuel additive may not be stringent, Isobutane oxidate, consisting of near equimolar proportions of crude TBA and TBHP, may be used as reaction feed thus lowering raw materials cost.

The reaction is driven to completion and the resulting crude DTBP - water mixture may be washed with water or slightly alkaline solution to remove soluble formates, aldehydes and ketones originally present in the feed; trace acid from the resin catalyst is also washed or neutralized in this step. The DTBP is then phase separated and dried (if required). Because the reactor effluent does not contain sulfuric acid, the caustic quench and neutralization is eliminated.

Project No. _____

(6) Test data and original references may be found on the following notebook pages 187436, 187437

Inventor (s) Signature	Date signed	Date of Conception
E. A. Hazbun E. A. Hazbun	10/13/81	
	Date signed	Date of Conception
Signature of Witness	Date signed	Date first disclosed to me
V. M. Chong	10/15/81	
Signature of Witness	Date signed	Date first disclosed to me
J. A. Cahill	10/15/81	

IMPORTANT

Chemical Company

Technical Suggestio

T. S. No. 81-7 Date Assigned 10/16/81

(1) Title of Invention: Improved Process for the Production of Ditertiary Butyl Peroxide

(2) - (3) - (4) - (5)

Such a process could yield DTBP of moderate purity at a transfer price in the 30 to 50¢/lb range depending on plant scale.

References

1. DTBP Production, R. L. Bobeck to B. Cramer, Aug. 1, 1979.
2. Initial Testing of DTBP as Octane Improver, J. M. DeJovine to J. A. Tarengelo, Jan. 14, 1981. Also U. S. Patent 2,378,341.

USE PEN OR TYPEWRITER - Suggested form for disclosure: (1) Title of invention, (2) State the practices prior to your invention, the problems and [illegible] connection with these prior practices, and how your invention solves these problems, (3) Your solution or invention - describe fully in prose, with [illegible] and structural formulae, when appropriate, (4) Give test results illustrating merits of your invention, (5) Describe principal advantages over prior practice realized by your invention, (6) List reference pages of laboratory notebook and the persons to whom the invention has been disclosed, (7) Names of inventor(s) witnesses are recorded on each sheet of the disclosure, (8) Cross out unused space, (9) Forward disclosure to Patent Department.

IMPORTANT

Project No. _____

(6) Test data and original references may be found on the following notebook pages 187437

Inventor (s) Signature	Date signed	Date of Conception
E. A. Hazbun E. A. Hazbun	10/13/81	
	Date signed	Date of Conception
Signature of Witness	Date signed	Date first disclosed to me
V. M. Chong	10/15/81	
Signature of Witness	Date signed	Date first disclosed to me
J. A. Cahill	10/15/81	

AtlanticRichfieldCompa[ny]

Research and Development

PROBLEM NO.

[SUB]JECT OF WORK Improved process for the production of ditertiary butyl peroxide.

T.S. 81-73 10/16/81 [illegible]

DATE

Witnessed E. McCaslin October 16, 1981

Ditertiary butyl peroxide (DTBP) is a specialty product used primarily as an organic peroxide catalyst or catalyst intermediate. It is commercially produced by the reaction of pure t-butanol (TBA) or isobutylene with hydrogen peroxide in the presence of concentrated sulfuric acid:

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The process requires refrigeration to control the reaction isotherm ($\Delta H = 98{,}000$ Btu/lb-mole) at or below 15°C thus reducing side reactions and TBA dehydration. The product mixture is quenched with excess water to promote phase separation. The organic phase, consisting of mostly DTBP, is washed with dilute alkali solution and the aqueous acid phase is usually neutralized and discarded.

When excess hydrogen peroxide is used in the above reaction, a mixture tert-butyl hydroperoxide and DTBP ~~are~~ is obtained.

SIGNATURE E. F. Harlan DATE 10/13/81 WORK CONTINUED ON PAGE 187435

WITNESSED AND UNDERSTOOD BY Victor M. Chong DATE 10/15/81

J. A. Cahill 10/15/81

AtlanticRichfieldCompany

Research and Development

PROBLEM NO.

OBJECT OF WORK Improved process for the production of ditertiary butyl peroxide

TS 81-73 10/16/81 cont'd.

DATE

Gellert E. McCabin - October 16, 1981

An alternative process, that has been experimentally demonstrated and recently evaluated (1), substitutes tert-butyl hydroperoxide for the hydrogen peroxide:

$$(CH_3)_3COH + (CH_3)_3COOH \xrightarrow[H_2SO_4]{70\%} (CH_3)_3COOC(CH_3)_3 + H_2O$$

This also requires refrigeration and acid neutralization. The estimated transfer price via this process at the 3 MM lbs/yr scale is $1.16/lb for pure DTBP.

Recent preliminary testing of DTBP as a cetane improver for diesel fuels (2) have shown the following encouraging results:

V %	Cetane #
0	44.8
3	56.8
5	67.8

Such a performance may create a large volume application for DTBP if it can be manufactured at low cost.

SIGNATURE E. F. Harlow DATE 10/13/81 WORK CONTINUED ON PAGE 187432

WITNESSED AND UNDERSTOOD BY Victor M. Chong DATE 10/15/81

J. A. Cooke 10/15/81

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WORK CONTINUED FROM PAGE 187435

AtlanticRichfieldCompa[ny]

Research and Development

PROBLEM NO.

OBJECT OF WORK Improved process for the production of ditertiary butyl peroxide.

TS 81-73 10/16/81 cont'd.

DATE

To allow continuous production at low cost, I propose that DTBP be produced by the reaction of TBHP and TBA over an ion exchange acid resin catalyst at a temperature sufficiently elevated to permit cooling with cooling tower water obviating the need for refrigeration.

Based on past experience with acid resin catalysts for MTBE and SBA production, these catalysts tend to yield more moderate reaction rates at higher selectivity than concentrated sulfuric or other mineral acid. A large pore size resin would be preferable in this instance. The reactor may be a multistage ~~&~~ packed bed with interstage cooling or a multitube heat exchange design.

Since the purity requirements for a diesel fuel additive may not be stringent, Isobutane oxidate, consisting of ~~about~~ near equimolar

Herbert E. McCarlin - October 16, 1981

SIGNATURE E. F. Harzbur DATE 10/14/81 WORK CONTINUED ON PAGE 187437

WITNESSED AND UNDERSTOOD BY Victor McChong DATE 10/15/81

J. A. Canale 10/15/81

WORK CONTINUED FROM PAGE 187436

AtlanticRichfieldCompany

Research and Development

PROBLEM NO.

OBJECT OF WORK Improved process for the production of ditertiary butyl peroxide

T.S. 81-73 10/16/81 Concluded

DATE

October 16, 1981 - Delbert E. Mc Cullin

proportions of crude TBA and TBHP, may be used as reaction feed thus lowering raw materials cost.

The reaction is driven to completion and the resulting crude DTBP-water mixture may be washed ~~in~~ with water or slightly alkaline solution to remove soluble formates, aldehydes and ketones originally present in the feed; trace acid from the resin catalyst is also washed or neutralized in this step. The DTBP is then phase separated and dried (if required). ~~Since~~ Because the reactor effluent does not contain sulfuric acid, the caustic quench and neutralization is eliminated.

Such a process could yield DTBP of moderate purity at a transfer price ~~below~~ in the 30 to 50 ¢/lb range depending on plant scale.

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1- DTBP Production, R.L. Bobeck to B. Cramer, Aug 1st, 1979

2- Initial testing of DTBP as Octane Improver, J.M. DeJovine to J.A. Tarengelo, Jan 14, 1981. Also US Patent 2,378,341

SIGNATURE E. F. Hazlewyn DATE 10/14/81 WORK CONTINUED ON PAGE

WITNESSED AND UNDERSTOOD BY Victor McChong DATE 10/15/81

J. A. Cobb 10/15/81